PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in the Manufacture of Surface Active Acylated Hydroxy Sulphonates

We, UNILEVER LIMITED, a Company registered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

agents. More particularly, it relates to the preparation of surface-active agents of the general formula R.COO.R¹.SO₃M where R is a monovalent hydrocarbon radical containing from 7 to 19 carbon atoms, R¹ is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms, and M is an alkali metal, by reaction of an acid of the formula R.COOH with a salt of the formula

HO.R¹.SO₃M.

Previously it has been proposed to carry out this reaction simply by heating together the two reactants at suitably elevated temperatures, as is described, for example, in B.I.O.S. Misc. Report 11, pages 31—2, for the cases where the acid is either oleic acid or a synthetic fatty acid, and R¹ is the radical

$-CH_2.CH_2-.$

This method suffers from certain disadvantages, namely, the excessive foaming which occurs during part of the reaction and which makes necessary the use of a reaction vessel very large relative to the volume of the reactants and the need for vigorous stirring in order to ensure adequate mixing of the phases of the reaction mixture. Further, the high temperatures at which the reaction mixtures must be maintained tend to cause discoloration of the products.

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It has now been found that the above reaction may be carried out more easily, quickly and completely by heating together the reactants in the presence of a reaction-promoting salt of a weak base and a strong inorganic or strong organic acid.

The disadvantages of the prior art method 45 may thus be over-come or reduced.

The present invention provides, therefore, a process for the preparation of surface-active agents of the formula R.COO.R1.SO3M where R is a monovalent hydrocarbon radical containing from 7 to 19 carbon atoms, R1 is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms and M is an alkali metal, by heating, under conditions such the water is removed, an acid of the formula R.COOH with a salt of the formula HO.R¹.SO₃M in the presence of a salt of a weak base and a strong inorganic or strong organic acid. By a strong acid is meant a compound whose acid dissociation constant is greater than 1×10^{-6} . By a weak base is meant a base weaker than the alkaline earth bases.

Compounds which have been found valuable in promoting the reaction are, for example, aluminium chloride, aluminium sulphate, alum, aluminium isethionate, basic aluminium acetate, aluminium dodecylbenzene sulphonate, aluminium dodecane sulphonate, aluminium acetylacetonate, stannous chloride, stannic chloride, zinc chloride, zinc sulphate, zinc isethionate, or hydrazine chloride. Aluminium salts are in general preferred. Salts of a weak base and an acid of the formula

HO.R¹.SO₃H,

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where R¹ is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms, are also valuable reaction-promoting

compounds.

The use of an isethionate is preferred in many cases as it contaminates the reaction product to a lesser extent. Where compounds 5 whose alkali metal salts yield surface-active agent such as, for example, a dodecylbenzene sulphonate or dodecyl sulphate are used, the resulting mixture may show enhanced surface-active properties.

In general, 0.01 to 10%, preferably 0.1 to 5%, of the reaction-promoting compounds by weight of the reaction mixture, is added, but greater or lesser amounts may be used.

The mode of action of the reaction-promot-15 ing compounds is not entirely understood but it is believed that they exert a catalytic influence. With their aid the reaction may give a yield of over 75% and in many cases a yield of over 90% and even of 96 to 100%, calculated on the salt HO.R¹SO₃M used, in a short time, for example one hour. The reaction, moreover, proceeds more smoothly and the disadvantages mentioned above do not arise.

It is preferred to carry out the reaction using as the compound of the formula R.COOH (hereinafter referred to as the first reactant) a compound in which the radical R is a saturated or unsaturated straight chain

30 primary alkyl group.

Examples of compounds which may be employed are fatty acids such as lauric acid, myristic acid, palmitic acid, stearic acid, oleic acid, acids derived from bone grease (mainly 35 C₁₆₋₁₈ fatty acids), acids derived from palm kernel oil (mainly C₁₂₋₁₈ fatty acids), acids derived from coconut oil (mainly C12-18 fatty acids), and acids derived from tallow (mainly a mixture of saturated and unsaturated C₁₆₋₁₈ fatty acids). The fatty acids used are

preferably pure. As the salts of the formula HO.R¹.SO₃M (hereinafter referred to as the second reactant) it is preferred to use a salt in which R1 is an 45 ethylene, methylethylene, dimethylethylene, propylene or butylene radical. R1 may also be a dialkylene ether radical, such as the radical —CH2CH2.O.CH2CH2—. Frequently, it will be convenient to use as the second reactant a compound which has been prepared by the reaction of an epoxide, for example, ethylene oxide, propylene oxide or butylene oxide, with sodium bisulphite.

Examples of compounds suitable for use as the second reactant are sodium isethionate, sodium methylisethionate, sodium dimethylisethionate and sodium 3 - hydroxypropanesul-

phonate.

When the second reactant is a compound in 60 which R1 is an alkylene radical containing from 2 to 8 carbon atoms, it is preferred to carry out the reaction using an excess of the fatty acid, for example 1.2 to 2 moles per mole of the second reactant. Excess fatty acid assists in maintaining the product in a

liquid form during the reaction, and also helps to prevent or reduce greatly the formation of foam. Furthermore, the products of such reaction are especially suitable for the manufacture of detergent tablets. The crude products formed may be either particularly or wholly neutralised by the addition of aqueous sodium carbonate or other alkali, such as sodium hydroxide or potassium carbonate, and the crude, partially neutralised or wholly neutralised products so formed may then be made directly into detergent compositions, for example, detergent tablets, by conventional methods of manufacture.

The reaction should be carried out with stirring in order to assist in homogenisation of the reaction mixture, but stirring of much less vigour is necessary in the presence of the reaction-promoting compounds of the invention than is the case when such compounds are not present. Mechanical agitation may be unnecessary where a gas, such as, for example, nitrogen, is bubbled through the reaction mixture at a moderate rate, or where the second reactant is added in the form of an aqueous solution. It is essential to remove the water which is formed in the course of the reaction or which may be present if either of the reactants is added as an aqueous solution. Reduced pressure may be employed if desired, particularly to remove water during the reaction, but reduced pressure is not an essention feature of the process. A small proportion of the first reactant may distill off with the water, and allowance should be made for 100 this when calculating the amount of first reactant to be used.

It has been found possible to carry out the reaction at lower temperatures in the presence of a reaction-promoting compound than in its 105 absence. For example, in the case of the reaction of C₈₋₂₉ fatty acids with sodium isethionate in the presence of one of the reaction-promoting compounds mentioned above, the reaction proceeds smoothly at 200° C. to 250° C., 110 whereas in the absence of such compound a temperature of 260° C. is essential for at least part of the process. It is desirable, however, in order to reduce the possibility of colour formation in the products, that an inert gas such 115 as oxygen-free nitrogen or carbon dioxide should be bubbled through the reaction vessel at such a rate as to maintain an inert atmosphere. It may also be desirable to bubble an inert gas through the reactants before they are 120 introduced into the reaction vessel in order to deoxygenate them. If very white products are desired it is preferred to use nitrogen that is freed from oxygen to a high degree, containing for example less than 100 p.p.m., and pre- 125 ferably less than 10 p.p.m., of oxygen.

The process according to the present invention may be carried out as a batch process, when all the reagents are present initially in the reaction vessel, or it may be modified to 130

form a continuous process by suitable selection of the conditions and of the apparatus. It may be advantageous to employ a high initial concentration of the reaction-promoting compound in the reaction mixture in order to facilitate the course of the reaction. This may be achieved by adding all the reaction-promoting compound to a small proportion, for instance, one-tenth, of the reactants, and then gradually adding the remainder of the reactants in approximately proportionate amounts after the reaction has commenced. It has also been found advantageous to add a small amount of the reaction product, for example, 10% by weight of the reaction mixture, during the initial stage of the reaction.

If desired, any excess of fatty acid in the reaction product may be removed, for example by distillation, preferably with the aid of an entraining agent such as toluene or steam or

under vacuum.

The following examples illustrate the invention:

EXAMPLE 1

400 g. of lauric acid (2 moles), 148 g. of sodium isethionate (1 mole) and 5.5 g. of one of the compounds mentioned in Table I below were placed in a flask equipped with a stirrer, thermometer, inert gas inlet and vapour outlet. The temperature was raised to 220° C. and maintained there for 1 hour. Throughout the reaction, the vapours from the reaction vessel were led through a condenser. The following amounts of sodium lauroyl isethionate, calculated on the sodium isethionate used, were obtained:

	TABLE I.	
	reaction-promoting compound	% yield
40	aluminium sulphate	100
	aluminium chloride	98—100
	aluminium isethionate	98—100
	hydrazine chloride	94
45	stannic chloride	93
	zinc sulphate	93
	stannous chloride	91
	zinc chloride	79
	zinc isethionate	78
50	basic aluminium acetate	77
	aluminium acetylacetonate	
	none	21
	The last result is included for	purposes of

comparison only.

Example 2.

412 g. of the fatty acids derived from coco-55 nut oil, hydrogenated to an iodine value of less than 3 and twice distilled, (2 moles), 148 g. of sodium isethionate (1 mole) and 5.6 g. of one of the compounds mentioned in Table II below were heated together under the same 60 conditions as described in Example 1. The following amounts of sodium fatty-acyl isethionate, calculated on the sodium isethionate used, were obtained:

TABLE II.		
reaction-promoting compound	% yield	65
aluminium isethionate	98	
aluminium chloride	91	
aluminium sulphate	89	
Example 3.		

247.2 g. of fatty acids derived from coconut oil (1.2 moles) were heated to 240° C. and 148 g. of sodium isethionate (1 mole) in the form of a 50% aqueous solution containing 4 g. of aluminium isethionate were added gradually over a period of 25 minutes. the reaction mixture being maintained at 240° C. during this time and for half an hour afterwards. The reaction product contained 98-100% of sodium fatty-acyl isethionate calculated on the sodium isethionate used.

EXAMPLE 4.

21.2 kg. of a sodium isethionate solution containing 85% of sodium isethionate and 15% of water, 34.5 kg. of hydrogenated twice distilled coconut oil fatty acids with an iodine value of 5.0, and 0.543 kg of $Al_2(SO_3)_3.18H_2O$ were heated together to 230° C. and kept at that temperature for $2\frac{1}{2}$ hours, being stirred with a stream of nitrogen containing less than 10 p.p.m. of oxygen at a rate of 1.5 cubic metres per hour. The reaction product was white and contained 95% of sodium fatty-acyl isethionate calculated on the sodium isethionate used.

EXAMPLE 5.

450 g. of lauric acid (2.25 moles), 243 g. of sodium 3 - hydroxypropane sulphonate (1.5 moles) and 7 g. of aluminium sulphate were heated together with stirring and in a current of nitrogen free from oxygen to a temperature of about 200° C. and kept there for 1 hour. The reaction product contained 75% of sodium 3 - lauroyloxypropane sulphonate calculated on the sodium 3 - hydroxypropane sulphonate used.

Example 6.

450 g. of lauric acid (2.25 moles), 243 g. of sodium 2 - methyl isethionate (1.5 mole) and 7 g. of aluminium sulphate were heated together under the conditions mentioned in Ex- 110 ample 5 for about 5 minutes. The reaction product contained 55% of sodium lauroy1 - 2methylisethionate calculated on the amount of sodium 2 - methyl isethionate used.

WHAT WE CLAIM IS:-1. A method of preparing a surface-active

agent of the general formula R.CO.O.R¹.SO₃M,

where R is a monovalent hydrocarbon radical containing from 7 to 19 carbon atoms, R1 is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms and 120 M is an alkali metal, in which an acid of the formula R.COOH is heated, under conditions such that water is removed, with a salt of the formula HO.R¹.SO₃M in the presence of a salt of a weak base with a strong inorganic 125 or strong organic acid.

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2. A method according to Claim 1 in which 0.1 to 10% by weight of the reaction mixture of the salt of a weak base with a strong inorganic or strong organic acid is used.

3. A method according to Claim 1 or 2 in which the reaction is carried out in the presence of an aluminium salt of a strong inor-

ganic or strong organic acid.

4. A method according to Claim 1 or 2 in 10 which the reaction is carried out in the presence of a salt of a weak base and an acid of the formula HO.R¹.SO₃H, where R¹ is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms.

5. A method according to any one of the preceding claims in which the reaction is carried out in the presence of aluminium ise-

thionate.

6. A method according to any one of Claims 1 to 3 in which the reaction is carried out in the presence of aluminium sulphate.

7. A method according to any one of the preceding claims in which the salt

HO.R¹.SO₃M

is sodium isethionate.

8. A method according to any one of the preceding Claims 1-6 in which the salt-HO.R¹.SO₃M is sodium 3 - hydroxypropane sulphonate.

9. A method according to any one of the preceding Claims 1-6 in which the salt HO.R¹.SO₃M is sodium methylisethionate.

10. A method according to any one of the preceding claims in which the compound 35 R.COOH is a fatty acid or a mixture of fatty acids.

11. A method according to Claim 10 in which the compound R.COOH is lauric acid.

12. A method according to Claim 10 in which the compound R.COOH is the mixture of fatty acids derived from coconut oil, which may be hydrogenated.

13. A method according to any one of the preceding claims in which the reaction is carried out at a temperature of 200 to 250°

C. in a stream of nitrogen.

14. A method according to Claim 13 in which the nitrogen contains less than 100 p.p.m. of oxygen.

15. A method of preparing a surface-active 50 agent of the general formula

R.CO.O.R¹.SO₃M

where R is a monovalent hydrocarbon radical containing from 7 to 19 carbon atoms, R1 is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms and M is an alkali metal, substantially as hereinbefore described, with particular reference to the Examples.

16. A surface-active agent of the general formula R.CO.O.R¹.SO₃M, where R is a monovalent hydrocarbon radical containing

from 7 to 19 carbon atoms, R1 is a divalent hydrocarbon or dialkylene ether radical containing from 2 to 8 carbon atoms and M is 65 an alkali metal, whenever prepared by a method according to any one of the preceding

claims.

UNILEVER LIMITED, R. JONAS, Agent for the Applicants.

PROVISIONAL SPECIFICATION

Improvements in the Manufacture of Surface-Active Acylated Hydroxy Sulphonates

We, Unilever Limited, a Company regi-70 stered under the laws of Great Britain, of Port Sunlight, in the County of Chester, England, do hereby declare this invention to be described in the following statement:—

This invention relates to surface-active 75 agents. More particularly, it relates to the preparation of surface-active agents of the general formula R.COO.R1.SO2M where R is a monovalent organic radical, R1 is a small divalent organic radical, and M is a cation, by reaction of an acid of the formula R.COOH with a compound of the formula HO.R¹.SO₃M.

Previously it has been proposed to carry out this reaction simply by heating together the two reactants at suitably elevated temperatures, as is described, for example, in B.I.O.S. Misc. Report 11, pages 31-2, for the cases where the acid is either oleic acid or a synthetic fatty acid, and R1 is the radical -CH₂.CH₂-.

This method suffers from certain disadvan-

tages, namely, the excessive foaming which occurs during part of the reaction and which makes necessary the use of a reaction vessel very large relative to the volume of the reactants, the need for vigorous stirring in order to ensure adequate mixing of the phases of the reaction mixture, and the necessity for reduced pressure to be maintained throughout the reaction. Further, the high temperatures at which the reaction mixtures must be main- 100 tained tend to cause discoloration of the products.

It has now been found that the above reaction may be carried out more easily and completely by heating together the reactants in 105 the presence of certain compounds which promote the reaction. The disadvantages of the prior art method may thus be overcome or reduced.

The present invention provides, therefore, a 110 process for the preparation of surface-active agents of the formula R.COO.R¹.SO₃M

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where R is a monovalent organic radical, R¹ is a small divalent organic radical, and M is a cation, by heating an acid of the formula R.COOH with a compound of the formula HO.R¹.SO₃M in the presence of a reactionpromoting compound as hereinafter described. Compounds which have been found valuable in promoting the reaction are those showing an acid reaction, in particular organic deriva-10 tives of strong inorganic acids, such as, for example, p-toluene sulphonic acid, beta-naphthalene sulphonic acid, dodecylbenzene sulphonic acid, dodecane sulphonic acid, isethionic acid or mono-dodecyl phosphoric acid. Other useful compounds include phosphoric acid and salts of strong acids with weak bases, such as, for example, aluminium isethionate, aluminium dodecylbenzene sulphonate, aluminium trichloride, stannous chloride or zinc chloride.

The use of isethionic acid is preferred in many cases as it does not contaminate the reaction product. Where compounds which on neutralisation yield surface-active agents, such as, for example, dodecylbenzene sulphonic acid or dodecane sulphonic acid, are used, the resulting mixture of the reaction product and the salt of the promoting substance may show enhanced surface-active properties.

In general, 0.01% to 10%, preferably 0.1% to 5%, of the reaction-promoting compound by weight of the reaction mixture, is added, but greater or lesser amounts may be used.

The mode of action of the reaction-promoting compounds is not entirely understood but it is believed that they exert a catalytic influence. With their aid the reaction may give a yield of over 80% and in many cases a yield of 98 to 100%, calculated on the salt HO.R¹SO₃M used, in a short time, for example one hour. The reaction, moreover, proceeds more smoothly and the disadvantages mentioned above do not arise. In certain cases a darkening of the reaction mixture is observed towards the end of the reaction, in particular with degrees of conversion above 90%, but this can easily be remedied by bleaching the reaction product, for example with hydrogen peroxide.

It is preferred to carry out the reaction using as the compound of the formula R.COOH (hereinafter referred to as the first reactant) a compound in which the radical R is a hydrocarbon radical of from about 7 to about 19 carbon atoms, more especially a saturated or unsaturated normal primary alkyl group. In other cases the radical R may contain or may consist of a ring system.

Examples of compounds which may be employed are fatty acids such as lauric acid, acids derived from bone grease (mainly C_{16-18} fatty acids), acids derived from palm kernel oil (mainly C_{12-18} fatty acids), acids derived from coconut oil (mainly C_{12-18} fatty acids), and acids derived from tallow (mainly a mixture of saturated and unsaturated

C_{16—18} fatty acids). As the compound of the formula

HO.R¹.SO₃M

(hereinafter referred to as the second reactant) it is preferred to use a compound in which R1 is a divalent hydrocarbon radical of 2-8 carbon atoms, especially an ethylene, propylene or butylene radical. R1 may also be a dialkylene ether radical, such as the radical -CH₂CH₂.O.CH₂CH₂-, or it may contain or consist of a ring system. Frequently, it will be convenient to use as the second reactant a compound which has been prepared by the reaction of an epoxide, for example, ethylene oxide, propylene oxide or butylene oxide, with sodium bisulphite. Examples of compounds suitable for use as the second reactant are sodium isethionate, sodium methyl isethionate and sodium dimethyl isethionate.

When the first reactant is a fatty acid of from 8-20 carbon atoms and the second reactant is a compound in which R1 is an alkylene radical of from 2 to 8 carbon atoms, it is preferred to carry out the reaction using an excess of the fatty acid, for example 1.1 to 2 moles per mole of the second reactant. Excess fatty acid assists in maintaining the product in a liquid form during the reaction, and also helps to prevent or reduce greatly the formation of foam. Furthermore, the products of such reactions are especially suitable for the manufacture of detergent tablets. The crude products formed may be either partially or wholly neutralised by the addition of aqueous sodium carbonate or other alkali, such as sodium hydroxide or potassium carbonate, and the crude, partially neutralised or wholly neutralised products so formed may then be made directly into detergent compositions, for example, detergent tablets, by conventional 105 methods of manufacture.

The reaction should be carried out with stirring in order to assist in homogenisation of the reaction mixture, but stirring of much less vigour is necessary in the presence of the 110 reaction-promoting compounds of the invention than is the case when such compounds are not present. Mechanical agitation may be unnecessary where a gas is bubbled through the reaction mixture at a moderate rate, or 115 where the second reactant is added in the form of an aqueous solution. It is essential to remove the water which is formed in the course of the reaction or which may be present if either of the reactants is added to an aqueous 120 solution. Reduced pressure may be employed if desired, particularly to remove water during the reaction, but reduced pressure is not an essential feature of the process. A small proportion of the first reactant may distill 125 off with the water, and allowance should be made for this when calculating the amount of first reactant to be used.

It has been found possible to carry out the reaction at lower temperatures in the presence of a reaction-promoting compound than in its absence. For example, in the case of the reaction of C₈₋₂₀ fatty acids with sodium isethionate in the presence of one of the reaction- promoting compounds mentioned above, the reaction proceeds smoothly at 200° C. to 235° C., whereas in the absence of such compound a temperature of 260° C. is essential for a least part of the process. It is desirable, however, in order to reduce the possibility of colour formation in the products, that an inert gas such as oxygen-free nitrogen or carbon dioxide should be bubbled through the reaction vessel at such a rate as to maintain an inert atmosphere. It may also be desirable to bubble an inert gas through the reactants before they are introduced into the reaction vessel, in order to deoxygenate them.

The process according to the present invention may be carried out as a batch process, when all the reagents are present initially in the reaction vessel, or it may be modified to form a continuous process by suitable selection of the conditions and of the apparatus. It may be advantageous to employ a high initial concentration of the reaction-promoting compound in the reaction mixture in order to facilitate the course of the reaction. This may be achieved by adding all the reaction-promoting compound to a small proportion, for instance, one-tenth, of the reactants, and then gradually adding the remainder of the reactants in approximately proportionate amounts after the reaction has commenced. It has also been found advantageous to add a small

ing the initial stage of the reaction.

If desired, any excess of fatty acid in the reaction product may be removed by distillation, preferably with the aid of an entraining agent such as toluene or steam.

amount of the reaction product, for example,

10% by weight of the reaction mixture, dur-

The following examples illustrate the invention:

Example 1.

400 g. of lauric acid (2 moles), 148 g. of sodium isethionate (1 mole) and 5.5 g. of one of the compounds mentioned in Table I below were placed in a flask equipped with a stirrer, thermometer, inert gas inlet and vapour outlet. The temperature was raised to 220° C. and maintained there for 1 hour. Throughout the reaction, the vapours from the reaction vessel were led through a condenser. The following amounts of sodium lauroyl isethionate,

calculated on the sodium isethionate used, were obtained:

TABLE I.		60
reaction promoting compound	% yield	
p-toluene sulphonic acid	98—10 0	
dodecane sulphonic acid	98—100	
beta-naphthalene sulphonic acid	98—100	
aluminium chloride	98—10 0	65
stannous chloride	91	
phosphoric acid	89	
mono-dodecyl phosphoric acid	87	
zinc chloride	79	
none	21	70
T		

Example 2

412 g. of the fatty acids derived from coconut oil, hydrogenated to an iodine value of less than 3, (2 moles), 148 g. of sodium isethionate (1 mole) and 5.6 g. of one of the compounds mentioned in Table II below were heated together under the same conditions as described in Example 1. The following amounts of sodium fatty-acyl isethionate, calculated on the sodium isethionate used, were obtained:

Table II.

reaction-promoting compound % yield dodecylbenzene sulphonic acid 98—100 isethionic acid 98—100 85 aluminium isethionate 98

EXAMPLE 3.

412 g. of fatty acids derived from coconut oil were heated in a flask to 240° C. 148 g. of sodium isethionate in the form of a 50% aqueous solution and 5.6 g. of p-toluene sulphonic acid were added gradually over a period of 25 minutes, the reaction mixture being maintained at 240° C. during this time and for half an hour afterwards. Fatty acids escaping together with water vapour were condensed and returned to the reaction mixture. The reaction product contained 98—100% sodium fatty-acyl isethionate calculated on the

sodium isethionate used. Example 4.

247.2 g. of fatty acids derived from coconut oil (1.2 moles) were heated to 240° C. and 148 g. of sodium isethionate (1 mole) in the form of a 50% aqueous solution and 4 g. of isethionic acid were added under the same conditions as those described in Example 3. The reaction product contained 98—100% sodium fatty-acyl isethionate calculated on the sodium isethionate used.

UNILEVER LIMITED, R. JONAS, Agent for the Applicants.

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